

**QUARTERLY PROGRESS REPORT NO. 8 ON THE DEVELOPMENT
OF THE PHYSICAL AND CHEMICAL PROPERTIES
OF THE SODIUM-POTASSIUM ALLOY**

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ABSTRACT

Physical and chemical properties of the sodium-potassium alloys are under study by this Laboratory. The status of active property measurements and all measured results obtained since the preceding Quarterly Report are presented; apparatuses for measurement of specific heat, thermal conductivity, and surface tension are described briefly.

The work which has been performed at the Mine Safety Appliances Company for this Laboratory under Contract No N6ori-146 was terminated on 24 June. Results of these investigations have been presented in various preliminary MSA reports, and a final report will be issued by that Laboratory.

PROBLEM STATUS

This problem was initiated in June 1946 upon the request of the Bureau of Ships. This is an interim report; work is continuing.

AUTHORIZATION

NRL Problem No. C11-02R (BuShips Project No. SRD 1399/48)

STATEMENT OF PROBLEM

The physical and chemical properties of liquid metals are to be investigated. The study to date has been concerned with the alkali metals.

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INTRODUCTION

The present report presents the status of active property measurements. Three apparatuses are described which are to be used in measurements of specific heat, thermal conductivity, and surface tension respectively. A preliminary reporting of the surface tension of sodium and potassium by the bubble pressure method is also included.

The work which has been performed at the Mine Safety Appliances Company for this Laboratory under Contract No. N6ori-146 was terminated on 24 June. Results of these investigations have been presented in various preliminary MSA reports, and a final report will be issued by that Laboratory.

THERMAL CONDUCTIVITY

General Remarks

A thermal conductivity study for the liquid alkali metals was made under government contract to Professor C. C. Bidwell of Lehigh University, Bethlehem, Pennsylvania. This thermal conductivity work was published in an earlier report of this series.¹

Comparable data from several independent sources has shed uncertainty on the overall accuracy of the results reported. In view of this, the relative importance of the thermal conductivity measurements has initiated further work along that line at this Laboratory and elsewhere.

The determination of conductivity coefficients of liquids with any degree of accuracy is extremely difficult, especially at high temperatures. The better methods employed by several investigators are far from standardized, and too, supposedly comparable results show for a large part poor

¹ Appendix B, NRL Report No. 3152, C. T. Ewing, R. S. Hartman, and H. B. Atkinson, Jr., "Quarterly progress report no. 4 on the measurement of the physical and chemical properties of the sodium-potassium alloy." (Restricted) July 1947

agreement. A portion of the disagreement evidently results from the high sensitivity of conductivity to traces of impurities, but certainly a large portion can be directly attributed to the practical difficulties of measurement. No attempt will be made at this time to analyze the available methods. For the present, it will suffice to say that the apparatus chosen for the work at NRL was developed from a comprehensive literature survey of the subject. The apparatus and method were determined after a careful consideration of several important points: (1) simplicity, (2) convection possibilities, (3) compensation of radial losses, (4) continuity of heat flow in sample rod, and (5) undesirable heat exchange (particularly with known heat source). In each case, an attempt was made to eliminate the questionable quantities which seem to be the inherent plague of conductivity measurements.

Description of Apparatus

The method is essentially one requiring unidirectional heat flow from a known heat source with measurable temperature gradients in the unknown. Radial heat losses are effectively eliminated by the conventional guard ring method with sil-o-cel as insulation. No attempt will be made to discuss the apparatus in great detail. Such a detailed description of apparatus and method will be presented when results are reported.

The stainless steel bar with sample container (approximately 4 cms in diameter and 50 cms long) is mounted on a stainless steel sink and supported inside a stainless steel vessel filled with sil-o-cel, the walls of the vessel serving as guard ring (Figure 1). The bar is hollowed to a wall thickness of 1/32 inch for a distance of 5 cm to form the specimen chamber. A small hole, representing a small fraction of the bar area, is drilled into the specimen chamber to permit filling. A stainless steel tube is welded at the top of this drilled hole to serve as an expansion reservoir. The liquid metals are to be distilled directly into the specimen chamber under high vacuum; a small section of nickel tubing at the top of the reservoir permits sealing off under vacuum.

The sample heating element is a helix of Pt10%Rh wire insulated and placed into grooves provided in the sample heater. Platinum leads extend through a uniform temperature region and out the top of the apparatus. A similar sample guard heater is placed above the sample heater to insure that the whole of the heat generated in the sample heater is transferred to the specimen. A pair of Pt-Pt10%Rh thermocouples are spaced between the sample heater and the sample guard heater for controlling heat losses. However, it is hoped that with adequate insulating, together with proper adjustment of the main and secondary guard ring heaters, that the use of the sample guard heater will become unnecessary.

Temperatures and temperature gradients in the sample rod and the specimen body are measured with No. 36 B&S Pt-Pt10%Rh thermocouples. The thermocouples embedded in the stainless steel container above and below the alloy body serve a multifold purpose. They provide control and estimation of radial losses, in addition to allowing an evaluation of conductivity coefficients relative to those of the stainless steel. The present intention is to measure (or have measured) independently the conductivity of the stainless

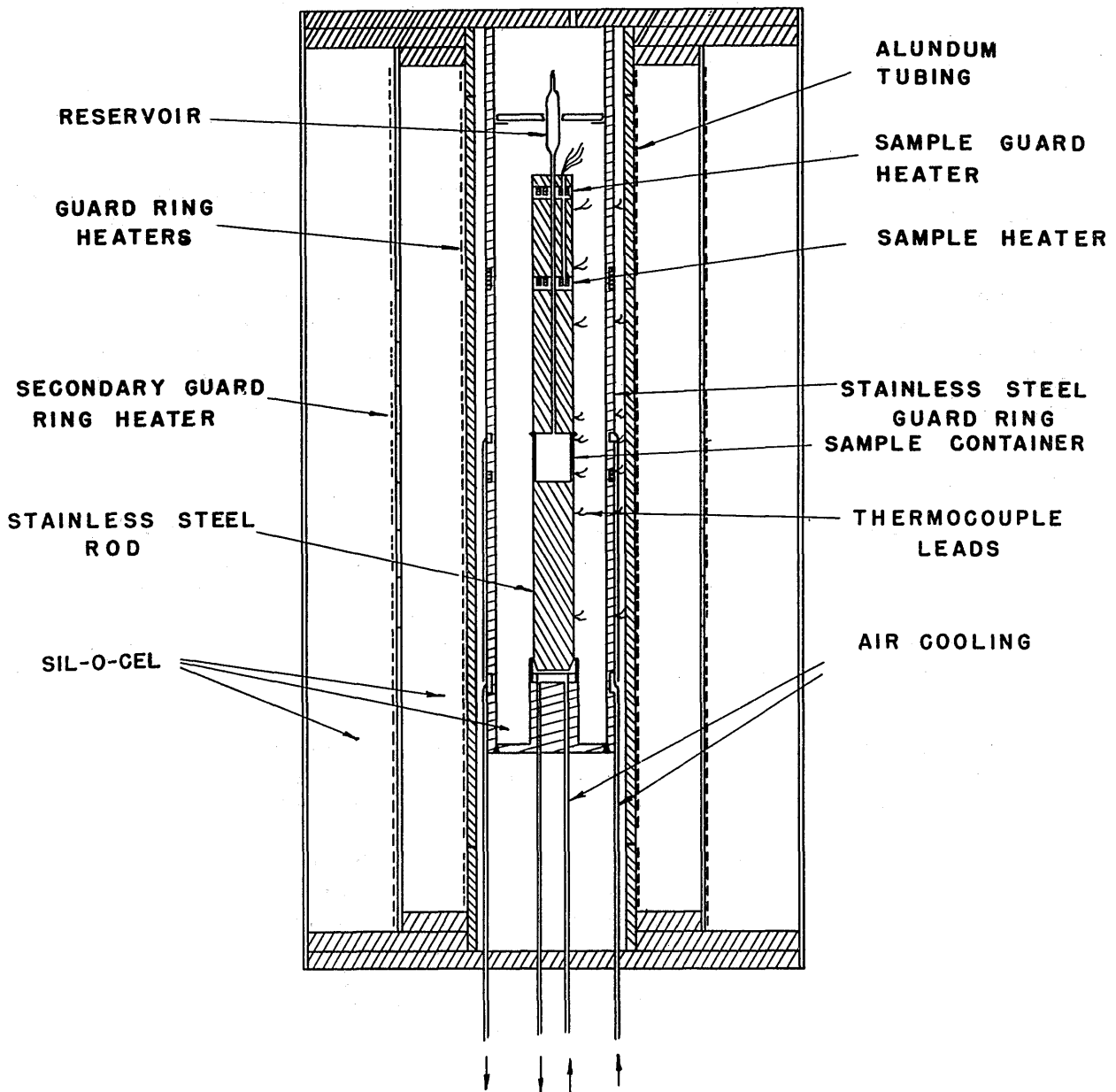


Figure 1 - Thermal conductivity apparatus

steel used in the sample rod. For control of radial losses, thermocouples of No. 26 B&S Pt-Pt10%Rh are provided in the guard ring opposite those in the sample bar.

The main furnace heater is wound uniformly in sections on an alundum tube surrounding the guard ring. Now, for effective control of the gradients in the guard ring, the radial losses from the guard ring should also

be compensated. For this purpose, a secondary guard ring heater is wound on a stainless steel cylinder and insulated radially both from the furnace heater and from the furnace casing with sil-o-cel. This heater is divided into a number of controlled sections for effective regulation of the radial losses in the guard ring in the measuring zone. All heaters are either of noninductive A-C windings or of D-C windings.

The guard ring heater is embedded in the guard ring opposite the sample heater. The heats withdrawn from the base of the guard ring and from the base of the sample bar are controlled independently by regulated air flow.

Conductivities of the liquid alloys appear to be near that of stainless steel, while the coefficients of the pure metals are reported to be somewhat higher. Therefore, to facilitate equalization of gradients in the region of the alloy specimen chamber, cooling is provided at the guard ring opposite the top of the chamber and heating opposite the base.

SPECIFIC HEAT

An apparatus for the measurement of specific heat modeled after one by Southard² was described in NRL Report No. C-3152. Measurements of sodium, potassium, and one alloy were made and found to be grossly in error. After a critical evaluation of the equipment, it was decided to modify the present specific heat apparatus to determine values for two alloys with an accuracy of 4 percent and also to design a new apparatus for further measurements with greater precision.

Many modifications (Figure 2) of the old apparatus were found necessary. The chromel-alumel furnace thermocouple was replaced with a Pt-Pt10%Rh thermocouple calibrated against a Bureau of Standards Pt-Pt10%Rh thermocouple. A sliding water gate was placed between the calorimeter receiver and furnace tube to prevent radiation from the furnace. Water thermostated at 30°C was circulated through the water gate. A test calibration with furnace at 450°C demonstrated the effectiveness of this radiation shield. The copper calorimeter receiver was fitted with self-closing lids to prevent radiation loss and hot air convection after the bucket has dropped into the calorimeter receiver. A constantan wire electric heater was wound around half of the outer surface of the receiver and protected by a tapered copper sleeve soldered to the receiver. Electrical calibration of the specific heat of the apparatus over the range 500-3000 calories input gave a precision of 1.5 percent to 0.13 percent indicating a precision of 6 calories. An insulated air thermostat was built around the Dewar flask and receiver and was set at 30°C with a variation of $\pm 0.04^\circ\text{C}$. The heat leak across the receiver amounts to 0.002°C per minute-degree. A new five-junction iron-constantan thermocouple was made and calibrated against a platinum resistance thermometer over the range 30° to 34°C with an accuracy of $\pm 0.001^\circ\text{C}$. New buckets or

² J. C. Southard, J. Amer. Chem. Soc. 63, 3147-50, November 1941

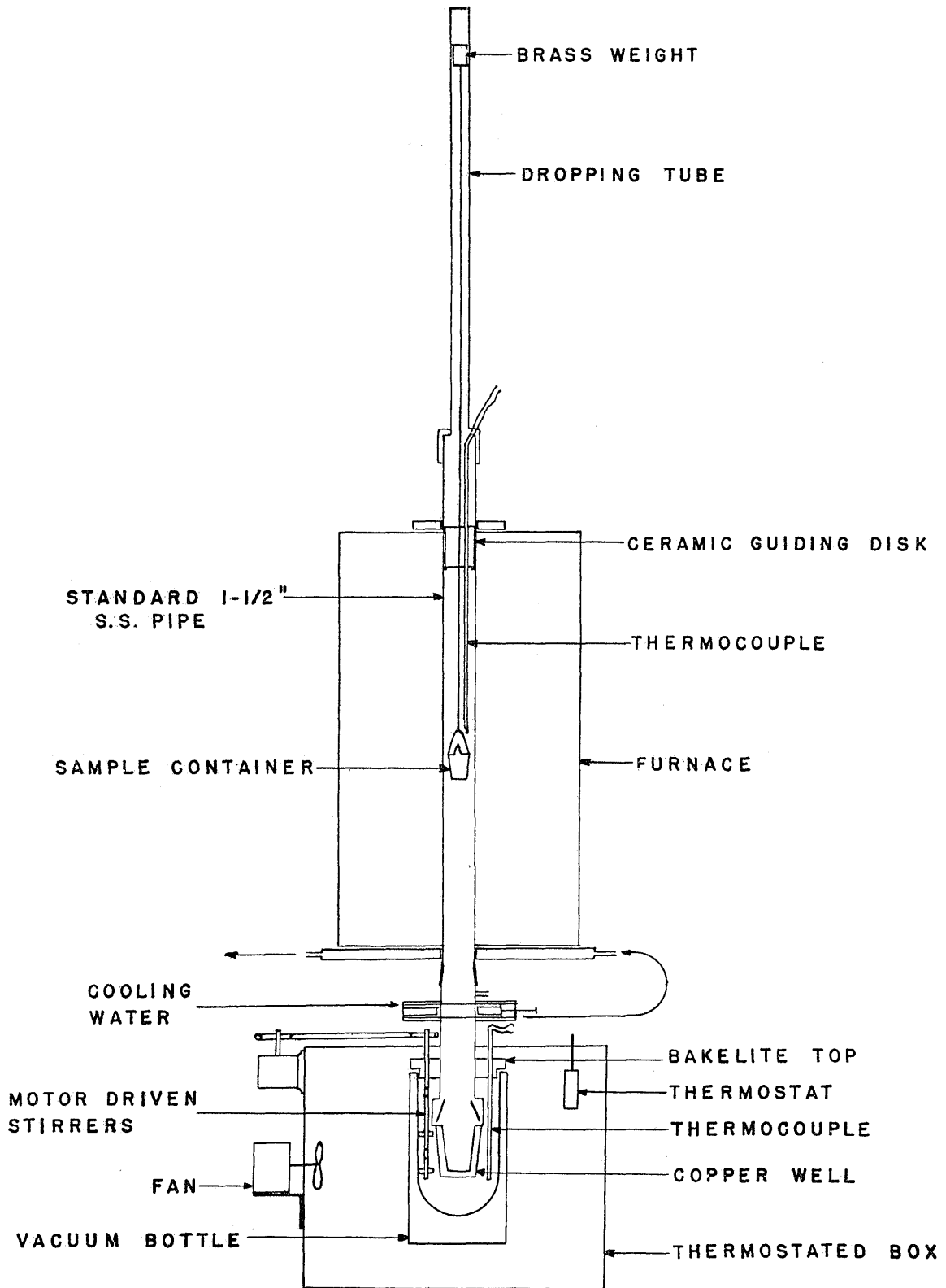


Figure 2 - Specific heat apparatus

alloy containers were made of electrolytic iron. The bucket walls were reduced to .020 inch thickness so that of the total heat capacity measured, alloy plus bucket, the alloy represents at least 50 percent of the total heat measured.

The present modified calorimeter has probable limits of error of the order of 2-4 percent on final measurements. Greatest sources of error are undoubtedly poor thermostating of the Dewar by air and the use of water as a calorimetric fluid with the accompanying stirring and evaporation errors. When present measurements are completed, it is planned to build a new calorimeter with a large block of copper as receiver, replacing the small copper receiver and water.

SURFACE TENSION

General Remarks

The maximum bubble pressure method is now being used for the determination of the surface tension of the sodium-potassium alloys. For the liquid metals, this method offers certain advantages over the capillary rise method. The interference which might be caused by the presence of any surface impurities is circumvented since each bubble produces a fresh surface. Another advantage is that the surface tension so measured is more independent of the contact angle between the liquid metal and the tube.

According to the bubble pressure method, a gas bubble is blown at the submerged end of a capillary tube which is dipped vertically downward a known distance in the liquid being tested. The pressure required to enlarge the bubble is measured and is a function of the surface tension. As the blown bubble becomes hemispherical, its radius of curvature diminishes and the pressure increases. Upon reaching a certain size, further growth results in a decrease of gas pressure within the bubble, gas rushes into the bubble from the tube, and the bubble breaks away to rise to the surface. If the bubble is sufficiently small, then its surface may be regarded as spherical, and the maximum pressure occurs when the bubble is a hemisphere whose radius corresponds to the radius of the capillary. The surface tension of the liquid may, therefore, be determined by the equation:

$$\gamma = \frac{r}{2} (p - h d g)$$

where

γ = Surface tension

r = Radius of capillary

P = Maximum pressure in the bubble as determined by measurement

h = Depth of end of capillary below the surface of the liquid

g = Gravity constant

d = Density of liquid being tested.

Description of Apparatus and Operations

The apparatus is diagramed in Figure 3. The differential manometer is filled with Octoil-S. Nitrogen (purified, as described in NRL Report No. C-3152, passing through a sintered glass filter containing sodium-potassium alloy) was used as static covering gas in the apparatus, and thus there was little likelihood of oxide formation when bubbles were blown.

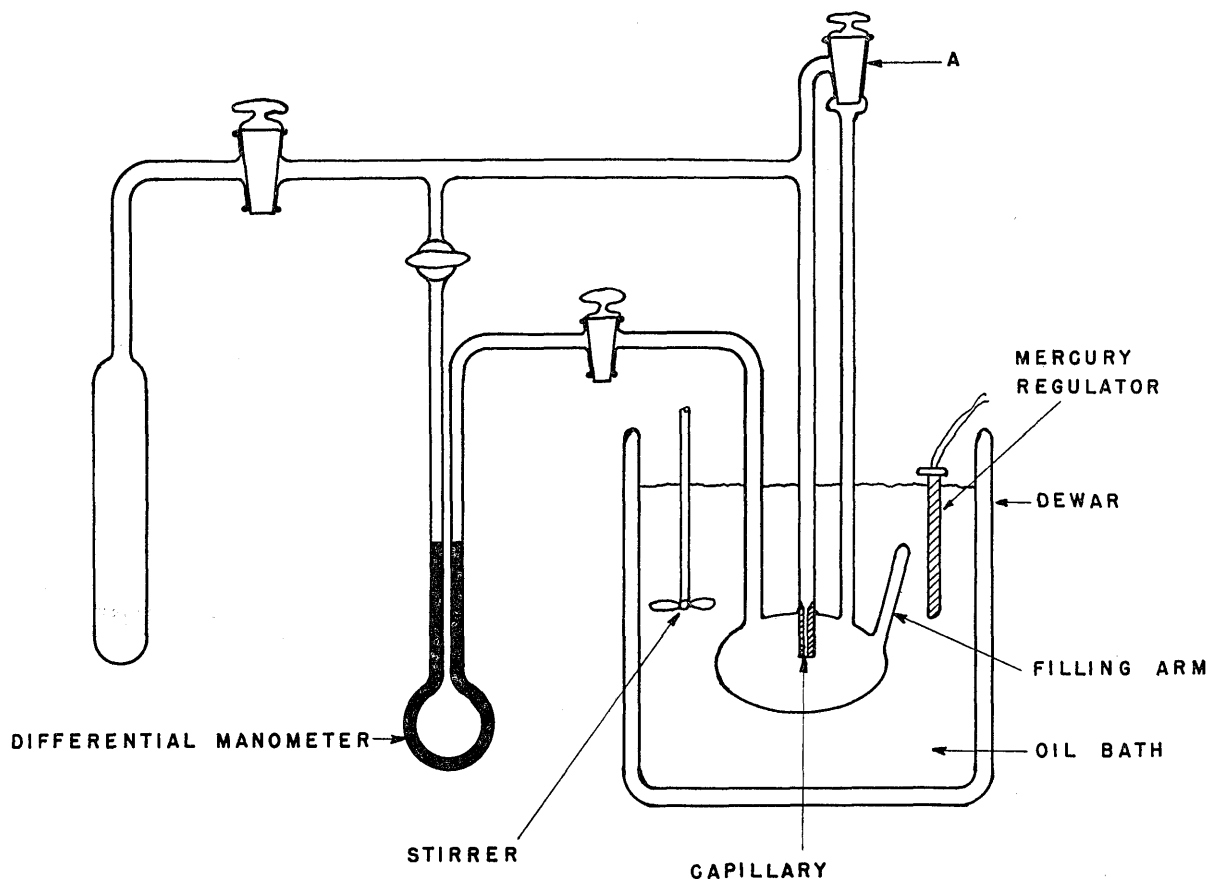


Figure 3 - Surface tension apparatus

The alloy was brought to equilibrium at some predetermined temperature by means of a silicone-oil bath. Stopcock (A) was then closed and the side bulb was warmed gently with a flame. The pressure developed, as shown by the manometer, was allowed to rise slowly until a maximum pressure was reached. Difference readings were then taken by means of a cathometer. By use of the equation already given, the surface tension of the liquid was calculated.

Experimental Results

Preliminary values for the surface tension of sodium and potassium are shown in the table. The values for sodium tend to substantiate the

magnitude of the values previously reported by the capillary rise method.³ It is noted that the values for potassium are of two definite magnitudes. This is believed to be due to a wetting phenomenon at the tip of the capillary. In such an event, the higher values are probably more correct. This point will be investigated by the substitution of capillary tips with thin walls. The surface tension studies are continuing.

SURFACE TENSION ALKALI METALS

Temperature	Sodium Surface Tension Dynes/cm	Potassium Surface Tension Dynes/cm
102.0	62.0, 71.0	26.6, 30.7
121.7	81.8, 70.8	61.6, 27.4
147.1	76.9, 69.7	63.6, 27.9
167.5	70.2, 67.4, 77.2, 80.5	65.8, 28.2
192	81.4, 70.3	64.0, 29.5
216	75.5	34.9

PURIFICATION STUDIES

The inherent difficulties involved in the study of contaminants present in the liquid metals are well known. An investigation is now underway to study solubilities of contaminants by means of radioactive tracers. It is hoped that the solubility-temperature curves for many of the contaminant salts and oxides can be obtained in this manner.

³ NRL Report No. 3287, C. T. Ewing, H. B. Atkinson, Jr., and T. K. Rice, "Quarterly progress report no. 7 on the measurements of the physical and chemical properties of the sodium-potassium alloy," (Unclassified) 24 May 1948.
